Training
Identification of Barbiturates

I. Introduction:

Barbiturates are screened and analyzed by GC/FID and subsequently confirmed by GC/MS. The samples are extracted by a simple solvent extraction procedure. However, an extraction procedure specific for non-volatile organic poisons can also be used (See Section IX).

If the sample is in a tablet or capsule form and has identifying imprints on it, the Identidex Imprint Identification program within the Micromedex Healthcare Series on the computer can be used to identify and verify the sample.

II. Reagents:

A.) Petroleum ether
B.) Methanol (GC solvent rinse)

III. Equipment:

A.) Analytical balance
B.) Magnifying microscope or magnifying glass
C.) 2 mL autosampler vials with Teflon caps
D.) GC/FID: HP 6890 or 5890 series
E.) GC/MS: HP 5890/5972 or HP 6890/5973 series
F.) Computer with Identidex Imprint Identification program.

IV. Procedure:

A.) Imprint Identification

1. Observe any imprint on tablet or capsule samples. Use a microscope or magnifying glass if necessary.
2. Record actual imprint, color, and shape of tablets or capsules in logbook.
3. On computer with Micromedex Healthcare Series, log onto the Identidex Imprint Identification page.
4. Enter the imprint code.
5. The program will generate a list of matches. Choose the first one on the list.
6. The identification, description and classification of the drug will appear. Read and verify that the computer's description matches your sample's description.
7. Print out the results, record results in logbook and file with the sample paperwork.

B.) Chromatography by GC/FID and GC/MS

1. If capsule or tablet contains no identifiable imprint code or if sample is in a powder form, the sample must be analyzed by GC/FID and GC/MS.
2. Place ¼ to ½ of tablet/capsule or 5 mg of powder sample into a 2 mL autosampler vial.
3. Add 1-2 mL of Petroleum ether to vial and cap.
4. Place on GC/FID autosampler and run with regular sequence (STD, BLK, Samples, STD).
5. GC/FID conditions are as follows:
   Method: EXP.M
   Oven:
      Initial Temp: 245°C
      Initial Time: 0.00 min.
      Rate: 10°/min.
      Final Temp: 290°C
      Run Time: 10 min.
      Max. Temp: 325°C
      Equilibration Time: 0.5 min.
   Inlet:
      Mode: split (35:1)
      Initial Temp: 250°C
      Pressure: 24.99 psi
      Gas Type: Helium
   Column:
      Capillary: HP-1 30m x 320um
      Initial Flow: 3.3 mL/min.
   Detector:
      Temp: 300°C
      Hydrogen Flow: 30.0 mL/min.
      Air Flow: 400 mL/min.
      Makeup Gas: Helium
6. Obtain chromatographs. If sample contains any barbiturates the instrument will detect a total ion peak with a retention time characteristic of that compound and will generate a report with accompanying chromatograph.
7. Check concentration to determine if dilutions are needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates that the sample may have to be reinjected.
8. Barbiturates are run with a different method on the GC/MS. This method analyzes for hydrocarbons and allows for a slower run time to detect early eluters.
9. Place the same sequence that ran on the GC/FID on the GC/MS autosampler.
10. GC/MS conditions are as follows:

Method: HYD.M
Oven:
Initial Temp: 130°C
Initial Time: 2.50 min.
Max. Temp: 325°C
Equilibration Time: 0.50 min.
Rate: 10°/min.
Final Temp: 280°C
Run Time: 30 min.
Inlet:
Mode: split (50:1)
Initial Temp: 250°C
Pressure: 31.65 psi
Gas Type: Helium
Column:
Capillary: HP-1MS 30m x 0.530mm
Max. Temp: 300°C
Initial Flow: 1.0 mL/min.

11. If any barbiturate is present in sample, the instrument will detect a total ion peak at its characteristic retention time and will generate a report along will accompanying chromatograph and spectra. The spectra will contain the identity if the peak and its ion abundance.

V. Results:

A.) Report a Barbiturate as positive when GC and GC/MS retention times and spectra match the standard Barbiturate. If a tablet or capsule imprint is identified in the Identidex Imprint Identification page, then GC/MS identification is sufficient.

B.) For the Identidex Imprint Identification procedure, record the identity of the sample in the logbook, as well as on the sample cards that came with the samples. Include date of analysis, results, and Chemist initials. Also, print out the results from the computer and file it with that sample number's paperwork.

C.) Record results of the GC/MS in logbook. Then transfer the results to appropriate sample cards that came with the actual samples. Be sure to include date of analysis, results, the number of tests performed per sample, and initials.

D.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.